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(21) International Application Number: PCT/US94/08496 (22) International Filing Date: 4 August 1994 (04.08.94) (30) Priority Data: 08/105,087 10 August 1993 (10.08.93) US (71) Applicant: E.I. DU PONT DE NEMOURS AND COMPANY [US/US]; 1007 Market Street, Wilmington, DE 19898 (US). (72) Inventor: OATHOUT, James, Marshall; 1008 Brenlan Court, Mt. Juliet, TN 37122 (US). (74) Agents: HANDLEY, Earl, L. et al.; E.I. du Pont de Nemours and Company, Legal/Patent Records Center, 1007 Market Street, Wilmington, DE 19898 (US).	(81) Designated States: CA, JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>	
(54) Title: HIGH ABSORBENCY CLEANROOM WIPES HAVING LOW PARTICLES (57) Abstract This application relates to a spunlaced fabric and a process for the production of a spunlaced fabric wherein the fabric is intended for use as a cleanroom wipe. Such cleanroom wipes preferably produce a low number of particles while having high absorbency. The fabric of the present invention essentially consists of a mixture of 25 to 65 wt. % cotton or rayon cellulose fiber and 35 to 75 wt. % polyester fiber. The fabric is further characterized in that it has a particle count of no greater than 18 million particles/m ² as measured by the Biaxial Shake (IES-RP-CC-004.2), an Intrinsic Absorbance of at least 5, and a Particle Sorbency Quotient of less than 55. The process of producing the fabric under the present invention is characterized by passing a web of 25 to 65 wt. % cotton or rayon cellulose fibers and 35 to 75 wt. % polyester fibers through a first series of water jets operating at a total impact energy of at least 10 X 10 ⁻³ horsepower-hour-pounds force/pound mass and a second series of water jets at the opposite side operating at a total impact energy of at least 20 X 10 ⁻³ horsepower-hour-pounds force/pound mass. In the process, the total impact energy of both sets of jets is at least 40 X 10 ⁻³ horsepower-hour-pounds force/pound mass.		

TITLEHIGH ABSORBENCY CLEANROOM WIPES HAVING LOW
PARTICLESFIELD OF THE INVENTION

5 This invention relates to cleanroom wipes which when used produce a low number of particles and have high absorbency, and to the process for the manufacture of such wipes. The wipes are manufactured by a spunlaced process i.e. a hydroentangling process.

BACKGROUND OF THE INVENTION:

10 Cleanroom wipes must produce a low number of particles when they are used, and it is desirable that they have relatively high absorbency rates and capacities. Wipes having all these desired properties have not been available commercially.

Processes for the manufacture of hydroentangled fibrous
15 webs are known in the art: see for example Evans U.S. Patent 3,485,706.

SUMMARY OF THE INVENTION:

The present invention is a spunlaced fabric consisting essentially of a mixture of 25 to 65 wt. % of a cellulose fiber selected from the group consisting of cotton and rayon, and 35 to 75 wt. %
20 polyester fiber, said fabric having a particle count no greater than 18 million particles/m² as measured by the Biaxial Shake (IES-RP-CC-004.2), an Intrinsic Absorbance of at least 5mL/g, and a Particle Sorbency Quotient (PSQ) of less than 55 million particle/liter sorbed.

The present invention is also a process for the production
25 of an absorbent, low particle-count spunlaced fabric which comprises:

a) passing a web consisting essentially of 25 to 65 wt. % of cellulose fibers selected from cotton and rayon, and 35 to 75 wt. % polyester fibers, supported on one of its two major surfaces by a foraminous screen under a series of water jets that traverse the
30 unsupported major surface of the web, said jets operating at a total impact energy of at least 10×10^{-3} horsepower-hour-pounds force/pounds mass thereby causing the cellulose fibers and the polyester fibers to entangle, and

b) passing the web of step a) supported on the second of
35 its two major surfaces by a foraminous surface under a series of water jets the traverse the unsupported major surface of the web, said jets operating at a total impact energy of at least 20×10^{-3} horsepower-

hour-pounds force/pound mass, thereby causing further entanglement of the cellulose fibers and the polyester fibers, and

the total impact energy of the jets of step (a) plus the jets of step (b) being at least 40×10^{-3} horsepower-hour-pounds

5 force/pound mass.

The process of the invention is preferably operated with the water jets using water at a temperature of at least about 30 degrees C.

BRIEF DESCRIPTION OF THE DRAWINGS

10 The invention will be better understood with reference to the following figures:

Fig. 1 is a schematic view of a continuous hydroentanglement process of the invention depicting belt and drum washers for water jetting both sides of a fabric web and a conventional squeeze roll for dewatering the resulting fabric following water jetting.

15 Fig. 2 is a schematic view of a continuous hydroentanglement process of the invention depicting belt and drum washers for water jetting both sides of a fabric web and a vacuum dewatering extractor for dewatering the resulting fabric following water jetting.

DETAILED DESCRIPTION OF THE INVENTION

Referring now to the figures, wherein like reference numerals represent like elements, schematic representations are shown of two continuous processes which are used in the invention. Fig. 1 depicts a continuous process wherein a web of fibers 10 (e.g., staple textile fibers of the invention) is air-laid onto a conveyer 12 having a mesh screen and conveyed towards a belt washer 14. The web is air-laid such that the textile staple fibers are supported by the mesh screen. Belt washer 14 contains a series of banks of water jets which treat the fiber web and entangle the textile staple fibers. Thereafter, the hydroentangled web is passed underneath another series of banks of water jets while it is supported on a patterning member of a drum washer 16. This patterning member consists of either 24, 40 or 100 mesh screens. The resulting fabric varies from apertured patterning with the 24 mesh screen to non-apertured non-patterning with the 100 mesh screen. Subsequently, the resulting spunlaced fabric is passed through a pair of squeeze rolls 18 to dewater the fabric. Thereafter, the

spunlaced fabric may be further treated by a padder 20, a dryer 22 and a slitter 24 before it is wound up on roll 26.

Fig. 2 is identical to Fig. 1, except that the squeeze rolls 18 have been replaced by a vacuum dewatering extractor 19. The vacuum extractor 19 is positioned between the drum washer 16 and the dryer 22.

As indicated above, the web is made up of staple textile fibers of the invention, in particular a mixture of cotton and polyester fibers or rayon and polyester fibers. Such webs may be produced by any conventional dry or wet method. Particularly preferred are the air-laid webs depicted in the Figures and produced according to U.S. Patent 3,797,074 (Zafiroglu), the entire contents of which are incorporated by reference herein.

During fabric manufacture, the fibrous web is subjected to jets of water delivered through closely-spaced small orifices. The jets impart to the web a total impact-energy product ("I x E") of at least 40×10^{-3} Horsepower-hour-pounds force/pounds mass ($\text{Hp-hr-lb}_f/\text{lb}_m$), preferably $60-80 \times 10^{-3} \text{ Hp-hr-lb}_f/\text{lb}_m$. Although this follows the general process of U.S. Patent 3,485,706 (Evans), the entire contents of which are incorporated herein by reference, the standard water-jet processing conditions are much less severe with a total I X E of about $20 \times 10^{-3} \text{ Hp-hr-lb}_f/\text{lb}_m$ or less. In addition, equipment of the general type described above, and mentioned in U.S. Patent 3,485,706 (Evans) and U.S. Patent 3,403,862 (Dworjanyyn), is suitable for the water-jet treatment. Further, an increase in jet water temperature appears to be advantageous, i.e., a 8 to 15 degree C increase over the normal room temperature (25 degrees C) water enhances the effect provided by the high impact-energy. The preferred temperature is above about 30 degrees C.

The energy-impact product delivered by the water jets impinging upon the fabric web is calculated from the following expressions, in which all units are listed in the "English" units in which the measurements reported herein were originally made so that the "I x E" ($\times 10^{-3}$) product was in horsepower-hour-pounds force per pounds mass.

$$I = 2PA'$$

$$E = PQ/wzs$$

wherein:

I is impact in lbs force

5 E is jet energy in horsepower-hours per pound mass

P is water supply pressure in pounds per square inch

A' is the apparent cross-sectional area in square inches and is
equal to about 0.6 A

A is cross-sectional area of the jet in square inches

10 Q is volumetric water flow in cubic inches per minute

w is web weight in ounces per square yard

z is web width in yards and

s is web speed in yards per minute.

The preferred cellulose fiber for use in the invention is
15 rayon, and the most preferred type of rayon is that made by the viscose
process.

The preferred fabric contains 30 to 60 % rayon, has a
particle count of no more than 5 million per square meter, and a PSQ of
no greater than 15.

20 TEST METHODS

The following test procedures were employed to
determine the various characteristics and properties reported below:

Wet particle counts were determined by the test methods
described in "Evaluating Wiping Materials Used in Cleanrooms and
25 Other Controlled Environments", Institute of Environmental Sciences,
IES-RP-CC-004.2 (August, 1992). The wet particle count (i.e., number of
particles suspended in water) is measured with a laser counter after the
fabric has been washed in water - either under conditions of minimum
stress (P_0) or after shaking in water for five minutes on a biaxial shaker
30 (BAS). Particle count is recorded as particles/ m^2 of fabric.

Absorptive capacity, either on a mass or area basis, is
measured according to the above-described IES-RP-CC-004.2. Stated
briefly, a weighed specimen of wiper is permitted unrestricted time and
mechanical stimulus to absorb all of the liquid it can from a pool of
35 water. The wiper is then removed from the pool, and allowed to drain
for 60 seconds, and the mass of the absorbed liquid that remains with
the wiper is determined. The data is reported in two ways: as an

intrinsic absorbency and an extrinsic absorbency. Intrinsic absorbency, A_i [mL/g], is defined as the volume of liquid sorbed per unit mass, while extrinsic absorbency, A_e [mL/m²], is the volume absorbed per unit area of wiper.

5 Absorption is also characterized by rate of absorption which is determined using a Gravimetric Absorbency Testing System (GATS), available from M/K Systems, Danvers, Massachusetts. In this test, a dry fabric specimen is placed onto a flat surface that is connected by a liquid bridge to a reservoir of water sitting on a top-loading
10 balance. As liquid is taken up by the fabric, the amount transferred from the reservoir to the fabric is recorded as a loss in weight at the balance. The corresponding time interval from test initiation is likewise recorded automatically. The uptake rate is obtained from the rate of change of the balance reading. Typical fabrics absorb liquid most
15 rapidly at the initiation of the test and more slowly as they reach their absorptive limit (absorptive capacity). The rate data reported herein is the rate of liquid uptake when the fabric has reached 50% of its total capacity (Rate @50% in g/g/s). Total capacity is reported herein as the weight of liquid sorbed by the fabric, expressed as a percentage based
20 on the sample weight.

Basis weight, [oz/yd²], is determined by measuring the mass of a 4 inch by 6 inch fabric sample according to the method described in INDA Standard Test IST 130.1 - 92, option 1.2.3, and reported as mass per unit area.

25 A single expression of two of the most important wiper parameters, absorbency and particles which can be removed, are described by the use of a Particle Sorbency Quotient, PSQ, which quantifies the number of particles introduced into an environment per one liter of water absorbed. Mathematically,

$$\begin{aligned} 30 \quad \text{PSQ} &= \text{Particles/Liter absorbed} \\ &= (\text{Particles/m}^2) / ((\text{mL/m}^2) / (\text{mL/L})) \\ &= \text{BAS} / (A_i / 1000) \end{aligned}$$

The values are expressed in million particles introduced per liter absorbed.

EXAMPLESEXAMPLE 1

In this Example, a spunlaced fabric of the invention was made with a mixture of rayon and polyester textile staple fibers in the form of an air-laid web. Commercially available "Dacron" polyester staple fibers (Type 612) from E. I. du Pont de Nemours and Co., Wilmington, Delaware, having a denier of 1.35 (1.5 dtex) and a length of 0.85 inch (2.16 cm) was combined with synthetic cellulosic staple fiber, a 100% Viscose rayon, code 1641, commercially available from Courtaulds Fibers, Inc., Axis, Alabama, having a denier of 1.8 (2.0 dtex) and a length of 1.125 inch (2.86 cm). The mixed staple fibers were air-laid according to the process described in U. S. Patent 3,797,074 (Zafiroglu). Based on the weight of the web, the web had a measured rayon content of about 31% wt.% and a polyester content of about 69 wt.%. 15

In a continuous operation, the web was supported on a smooth foraminous screen (approximately 76 mesh) such that the bottom side of the web was in contact with the screen. Thereafter, the web was passed along at a belt washer speed of 18 yds/min (16.5 m/min) and then passed underneath a series of banks of belt washer jets under conditions as shown in Table I. The water used for the jets was once-through water that had not been recirculated. In a continuous operation, the web was wrapped around a drum washer over a 40 mesh screen so that the other side of the web (i.e., side contacting the belt washer in that treatment) could be passed underneath a series of banks of drum washer jets under conditions as shown in Table II. Following the drum washer treatment, the spunlaced fabric was dewatered using a vacuum dewatering extractor, dried and wound up. It should be noted that the wind-up speed of the fabric was 20 yds/min (18.3 m/min) and this value was used to calculate the "I x E" product in the Tables below. 20 25 30

7

TABLE I

Belt Washer Treatment

	Jet	Orifice Diameter	# of Jets per	Pressure	I x E	
		Water				
5	No.	inch (mm)	inch (cm)	psi	Hp-hr-lbf/lb _m	
		Gal/min				
					X 10 ⁺³	
10	1	0.005(0.127)	40(15.7)	100	0.01	9
	2	0.005(0.127)	40(15.7)	500	0.34	20
	3	0.005(0.127)	40(15.7)	1000	1.90	28
	4	0.005(0.127)	40(15.7)	1500	5.23	34
	5	0.005(0.127)	40(15.7)	1915	9.63	39
	6	0.005(0.127)	40(15.7)	2000	10.73	39
Total					27.84	169

TABLE II

Drum Washer Treatment

Jet	Orifice Diameter		# of Jets per	Pressure	I x E	
	Water					
20	No.	inch (mm)	inch (cm)	psi	Hp-hr-lb _f /lb _m	
		Gal/min			X 10 ⁺³	
25	1	0.005(0.127)	60(23.6)	450	0.39	28
	2	0.005(0.127)	40(15.7)	800	1.09	25
	3	0.005(0.127)	60(23.6)	1200	4.49	46
	4	0.005(0.127)	60(23.6)	1500	7.84	51
	5	0.005(0.127)	60(23.6)	1915	14.44	48
	6	0.005(0.127)	60(23.6)	2000	16.10	59
Total					44.35	257

The fabric was tested for absorption and wet particle release and generation under conditions of minimum stress or after shaking for five minutes on a biaxial shaker as discussed above. The results are tabulated below in Table III and can be compared to results for standard spunlaced product and several competitive wipe fabrics which are found in Table IV. The substantial reduction in particle generation is attributed to the effect of the higher-than-normal impact-energy and the somewhat elevated jet-water temperature which is thought to physically remove and at least partially dissolve the

removed particles. The inventive fabric has much lower particle generation compared to standard spunlaced rayon/polyester products, Comparative Examples A and B, or other competitive wiper products of rayon or cotton, Comparative Examples D and E. In fact, the
 5 inventive fabric is quite comparable to a the TEXWIPE TX1010 knit polyester, Comparative Example C, a high quality cleanroom wipe, in terms of low particle generation but much superior in absorption.

Examples 2 to 7 were prepared similarly to Example 1 with any changes, e.g. I X E, water-jet temperature, fiber content, noted
 10 in Tables III and V along with the results of testing for absorption and particle generation. Example 2 was run at conditions not much different from Example 1 with equally good results. Example 3, although prepared at a I X E of 45×10^{-3} compared to about an I X E value about 72×10^{-3} for Examples 1 and 2 and possessing a higher
 15 rayon content, is still much improved over the standard spunlaced product, Comparative Examples A and B, and the competitive products, Comparative Examples C, D, and E.

Examples 4 to 7 compares rayon to cotton (pre-opened, bleached, and scoured cotton staple of approximately 1 inch (2.54 cm),
 20 coded 563004, obtained from Veratec, Inc., Walpole, Maine, a division of International Paper) at 50 wt.% cellulosic to polyester content and shows that there is essentially no effect on particle generation when varying the mesh of the drum screen from 24 which gives an apertured, patterned product to 100 mesh which gives a non-apertured, non-
 25 patterned product. Although not improved to the same extent as the rayon, the cotton/polyester product of the invention is substantially improved over the competitive cotton product generating much, much lower number of particles generated in testing.

TABLE III

30	Example	1	2	3
	Composition	31% rayon	37% rayon	60% rayon
	Water Temperature, °C	33	40	25
	Drum Mesh	40	40	40
	I X E (Belt)	27.8	27.6	22.5
35	I X E (Drum)	44.3	44.1	22.5
	I X E - Total	72.1	71.7	45.0
	Dewatering	Vacuum	Vacuum	Squeeze

TABLE III (cont'd)

	Intrinsic Absorbance, A_i , mL/g	6.1	5.8	6.4
	Extrinsic Absorbance, A_e , mL/m ²	412	369	427
5	Absorbent Capacity, %	621	587	490
	Absorbent Rate, @50% Capacity g/g/s	0.20	0.13	0.27
	Basis Weight, oz/yd ²	2.01	1.91	2.21
10	Particle Count, P_0 , 10 ⁶ /m ²	1.5	2.1	7.9
	Particle Count, BAS, 10 ⁶ /m ²	0.5	1.7	4.3
	PSQ, 10 ⁶ particles/L	1.2	4.7	10.1

TABLE IV

COMPARATIVE EXAMPLES

15		STANDARD SPUNLACED		COMPETITIVE		
	EXAMPLE	A	B	C	D	E
		SONTARA	SONTARA	TEXWIPE	BEMCOT	BERKSHIRE
		STYLE 8423	STYLE 8425	TX1010	CT-8	
		SPECWIPE-1				
20	Composition	70%	50%	100%	100%	
		100%				
		rayon	rayon	polyester	rayon	
		cotton				
				knit		
25	Basis Weight	2.3	1.8	4.2	0.92	4.3
	oz/yd ²					
	A _i , mL/g	6.63	6.9	2.0	9.0	1.62
	A _e , mL/m ²	508	417	226	280	271
	I X E - Total	21.1	26.1	NA	NA	NA
30	Particle Count,	3.3	81	2.3	33	34
	P ₀ , 10 ⁶ /m ²					
	Particle Count,	32	18	4.1	193	301
	BAS, 10 ⁶ /m ²					
	PSQ,	63	43	18	689	1111
35	(10 ⁶ part)/L					

10

TABLE V

Example	4	5	6	7
Composition	50% cotton	50% cotton	50% rayon	50% rayon
Water Temperature, degrees C	25	25	25	25
5 Drum Mesh	24	100	100	24
I X E (Belt)	20	20	20	20
I X E (Drum)	20	20	20	20
I X E - Total	40	40	40	40
10 Dewatering	Squeeze	Squeeze	Squeeze	Squeeze
Intrinsic Absorbance, A_i , mL/g	5.8	5.5	6.0	6.4
Extrinsic Absorbance, A_e , mL/m ²	342	321	369	393
15 Absorbent Capacity, %	501	517	523	513
Absorbent Rate, @50% Absorption g/g/s	0.23	0.23	0.22	0.20
Basis Weight, oz/yd ²	1.75	1.80	1.77	1.76
Particle Count, P_o , 10 ⁶ /m ²	5.4	7.0	8.6	14
20 Particle Count, BAS, 10 ⁶ /m ²		11.7	14.4	3.0 4.2
PSQ, 10 ⁶ particles/L	34	45	8	11

Claims:

1. A spunlaced fabric consisting essentially of a mixture of 25 to 65 wt. % of a cellulose fiber selected from the group consisting of cotton and rayon, and 35 to 75 wt. % polyester fiber, said fabric
5 having a particle count no greater than 18 million particles/m² as measured by the Biaxial Shake (IES-RP-CC-004.2), an Intrinsic Absorbance of at least 5, and a Particle Sorbency Quotient of less than 55.
2. The spunlaced fabric of claim 1 in which the cellulose
10 fiber is rayon made by the viscose process.
3. The spunlaced fabric of claim 1 in which the cellulose fiber is rayon, the rayon is present in the amount of between 30 and 60 %, the fabric having a particle count no more than 5 million/m² and a Particle Sorbency Quotient of no greater than 15.
- 15 4. The spunlaced fabric of claim 3 in which the rayon is made by the viscose process.
5. A process for the production of an absorbent, low particle-count spunlaced fabric which comprises:
 - a) passing a web consisting essentially of 25 to 65 wt. % of
20 cellulose fibers selected from cotton and rayon, and 35 to 75 wt. % polyester fibers, supported on one of its two major surfaces by a foraminous screen under a series of water jets that traverse the unsupported major surface of the web, said jets operating at a total impact energy of at least 10×10^{-3} horsepower-hour-pounds
25 force/pounds mass thereby causing the cellulose fibers and the polyester fibers to entangle, and
 - b) passing the web of step a) supported on the second of its two major surfaces by a foraminous surface under a series of water jets that traverse the unsupported major surface of the web, said jets
30 operating at a total impact energy of at least 20×10^{-3} horsepower-hour-pounds force/pound mass, thereby causing further entanglement of the cellulose fibers and the polyester fibers, and
the total impact energy of the jets of step (a) plus the jets of step (b) being at least 40×10^{-3} horsepower-hour-pounds
35 force/pound mass.
6. The process of claim 5 in which the water temperature is at least about 30 degrees C.

FIG. 1

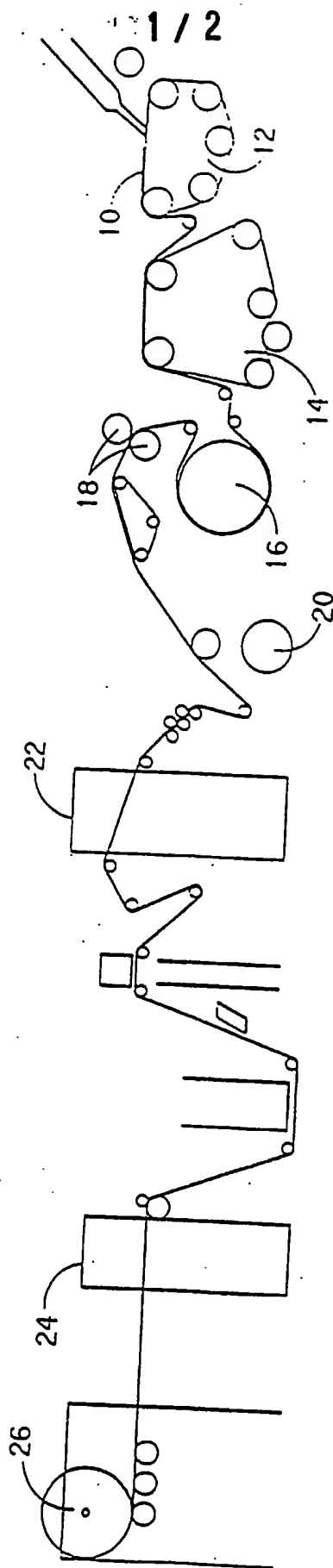
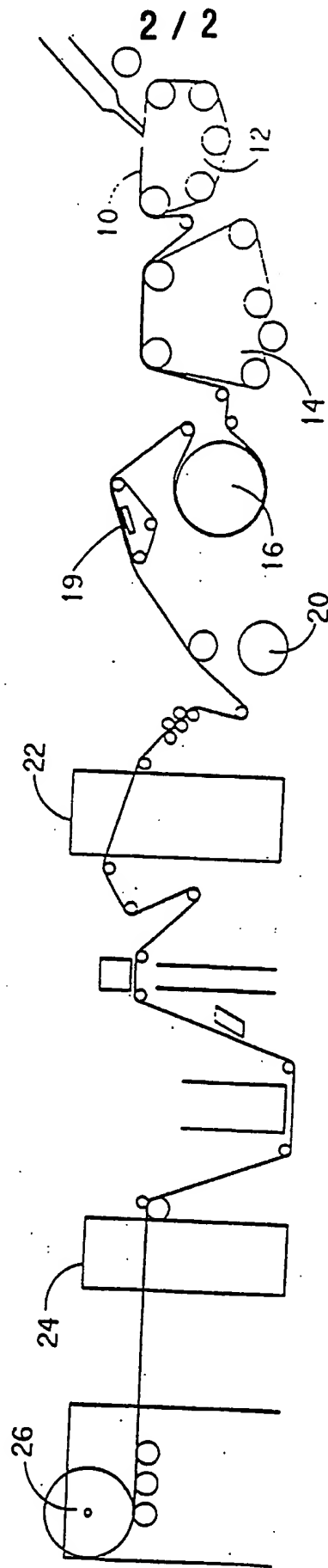


FIG. 2



INTERNATIONAL SEARCH REPORT

Int. Patent Application No

PCT/US 94/08496

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 6 D04H1/44 A47L13/16

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
 IPC 6 D04H A47L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,4 693 922 (BUYOFSKY ET AL.) 15 September 1987 see column 2, line 9 - column 5, line 13 ----	1
A	US,A,5 093 190 (KWOK ET AL.) 3 February 1992 see column 1, line 14 ----	
A	US,A,3 485 709 (EVANS ET AL.) 23 December 1969 see column 2, line 61 - column 5, line 15 -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

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